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| 13. ABSTRACT (Maximum 200 words)<br><br>Multilayer polyimide(PI) films were successfully fabricated using the Langmuir-Blodgett(LB) technique. These films were studied in several ways relevant to their use as liquid crystal alignment layers. (1) The influence of dipping speed and creep time on the orientational order of PI-LB films was investigated by means of birefringence measurements. (2) For comparison, we measured the pretilt angles and polar anchoring strengths of liquid crystal(LC) cells assembled with PI-LB films and rubbed PI-LB films as alignment layers. (3) The anchoring directin of the liquid crystal was found to be solely determined by the dipping direction of uppermost PI-LB layer, regardless of the dipping direction of deeper layers. (4) Combining results of the above studies with measurements of pretransitional birefringence(above the isotropic to nematic(I→N) transition) and observations of the growth of the alignment texture just below the nematic-isotropic transition, we draw the conclusion that the range of interaction between the PI-LB film and LC molecules is quite short( $\sim 4.5\text{ \AA}$ ) and that the alignment mechanism is epitaxial. Therefore, anisotropic short range molecular interactions are responsible for the alignment of the first liquid crystal layer. (5) From studies of PI-LB films deposited perpendicular to the rubbing direction of underlying spin coated PI films, we also found evidence, as expected, that the grooves induced by the rubbing process are not decisive for LC alignment on a rubbed polymer surface. |  |   |  |                                  |
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Investigations on Langmuir-Blodgett films as  
alignment layers for liquid crystals

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## Abstract

Multilayer polyimide(PI) films were successfully fabricated using the Langmuir-Blodgett(LB) technique. These films were studied in several ways relevant to their use as liquid crystal alignment layers.

(1) The influence of dipping speed and creep time on the orientational order of PI-LB films was investigated by means of birefringence measurements. (2) For comparison, we measured the pretilt angles and polar anchoring strengths of liquid crystal(LC) cells assembled with PI-LB films and rubbed PI-LB films as alignment layers. (3)The anchoring direction of the liquid crystal was found to be solely determined by the dipping direction of uppermost PI-LB layer, regardless of the dipping direction of deeper layers. (4) Combining results of the above studies with measurements of pretransitional birefringence(above the isotropic to nematic(I→N) transition) and observations of the growth of the alignment texture just below the nematic-isotropic transition, we draw the conclusion that the range of interaction between the PI-LB film and LC molecules is quite short( $\sim 4.5\text{\AA}$ ) and that the alignment mechanism is epitaxial. Therefore, anisotropic short range molecular interactions are

responsible for the alignment of the first liquid crystal layer. (5)  
From studies of PI-LB films deposited perpendicular to the rubbing  
direction of underlying spin coated PI films, we also found evidence,  
as expected, that the grooves induced by the rubbing process are  
not decisive for LC alignment on a rubbed polymer surface.

## I. Introduction

The wide application of flat panel displays using liquid crystals(LCDs) has stimulated extensive study of alignment<sup>[1-3]</sup>. Conventional processes for forming homogenous alignment layers include a rubbed polymer coating and oblique evaporation of SiO<sub>x</sub><sup>[2]</sup>. Although the oblique evaporation method has the advantage of giving the liquid crystal molecules a prescribed pretilt angle, the deposition process is quite complicated and it is difficult obtain a large uniform aligning area. The rubbing process also has several serious shortcomings<sup>[4,5]</sup>. Static electricity generated by rubbing causes flicker during operation of LCDs. Furthermore, the delicate switching elements on the surface, for example thin film transistors, can be destroyed by the rubbing process and rubbing can introduce dust particles which can degrade the performance of LCDs. For these reasons, researchers have tried to develop alternative methods of alignment. Ordered polyimide Langmuir-Blodgett(PI-LB) films have recently been shown to have enormous promise as LCD alignment layers<sup>[6-11]</sup>. It is now clear that, LB films with their well-controlled architecture, large-area capability and dust free character are contenders for the

alignment layers of future LCDs. Due to this potential application in the LCD industry, we need to know the mechanism of alignment and how to control the important parameters for display, for example anchoring strength and pretilt angle.

In this paper, we present our preliminary results on PI-LB film alignment layers. Orientational order of PI-LB films was investigated by means of the birefringence measurements on films prepared with different dipping speeds and creep times. The pretilt angle and the temperature dependence of the polar anchoring strength of liquid crystal cells assembled with PI-LB alignment films and their rubbed counterparts were also measured. We have also made the first study of the pretransitional birefringence of LC cells with PI-LB alignment films. Based on our experimental results, the LC alignment mechanism on PI-LB films is suggested. Finally, we bring in to focus the advantages and problems associated with PI-LB films as alignment layers. Possible solutions for the existing problems are suggested.

## II. Experimental

### 1. Preparation of multilayer PI-LB films:

Fig.1 shows the flow chart of the PI-LB preparation. LB films were prepared in a  $70\text{cm} \times 15\text{cm}$  Lauda trough. Due to the non-amphiphilic nature of the PI, we first mixed poly[4,4'-oxydiphenylene] pyromellitic acid (I) with N,N' dimethylhexadecylamine (II) in the molar ratio of 1:2 in an organic solvent of N,N-dimethylacetamide and benzene(1:1 in a molar ratio) to form a poly[4,4'-oxydiphenylene] pyromellitic acid salt(III).  $100\mu\text{l}$  of (III) was then spread on deionized water at  $20^\circ\text{C}$ . After waiting for 10 minutes, the monolayer was compressed at a speed of  $1.8\text{cm/min}$ . The value of the co-area obtained was  $110\text{\AA}^2$ . Y-type LB films of (III) were transferred onto the indium-tin-oxide(ITO) coated patterned glass by vertical dipping at a surface pressure of  $25\text{mN/m}$  and dipping speed of  $1\text{mm/min}$  for the first round trip and  $10\text{mm/min}$  for subsequent dipping. Films of (III) were immersed overnight in a mixed organic solvent of acetic anhydride, pyridine and benzene(1:1:3) for imidization to obtain polyimide(IV) LB(PI-LB) films.

## 2. Measurements of the anchoring strength, pretilt angle and optical retardation:

The liquid crystal cells were made with two identical ITO patterned glass substrates coated with PI-LB films. We assembled the cells with the dipping direction of the last LB layer antiparallel to each other. The thickness( $\sim 50\mu m$ ) of each cell was determined by the optical interference method ( $\pm 0.5\mu m$ ) before the cells were filled with 4-cyano-4'-n-pentybiphenyl(5CB) liquid crystal by the capillary method. These cells were heated to  $80^{\circ}C$ (above the nematic-isotropic clearing point) then slowly cooled down to room temperature to get rid of the flow alignment effect induced by filling the cell. 5CB was purchased from BDH chemicals and used without further purification.

The optical retardation of the PI-LB film was measured by ellipsometry with a resolution of  $0.01^{\circ}$ . The standard crystal rotation method<sup>[12]</sup> was used to determine the pretilt angle. To measure the polar anchoring strength, we adopted Yokoyama and VanSprang's high field method<sup>[13,14]</sup>, which requires simultaneous measurements of the voltage dependence of the birefringence and capacitance of a

homogeneously aligned nematic LC cell. The schematic diagram for the optical phase retardation and capacitance measurement apparatus is shown in Fig.2.

For applied voltages,  $V$ , larger than 6 times the Freedericksz threshold voltage,  $V_{th}$ , the following relation should be accurate<sup>[13]</sup>:

$$R/R_0 = I_0/CV - 2d_e/d_0 \quad (1)$$

where the constant  $I_0$  is related to material parameters.  $R_0$  is equal to  $2\pi d_0(n_e - n_o)/\lambda$ .  $d_e$  is called the surface extrapolation length. The polar anchoring strength,  $W_0$ , is directly related to  $d_e$ . In particular, for the Rapini-Papoulier surface potential,  $\frac{1}{2}W_0 \sin^2(\theta - \theta_e)$ , we have a simple relationship  $W_0 = K_1/d_e$ , where  $K_1$  is the splay elastic constant of the LC which was measured separately.  $C$  and  $d_0$  are the capacitance and thickness of the LC cell respectively.  $n_0$  and  $n_e$  are the ordinary and extraordinary refractive indexes of the LC. Temperature controlled to  $\pm 5\text{mK}$  over 10hrs. For the measurement of the pretransitional birefringence, the temperature was swept at rate of  $0.18^\circ\text{C}/\text{hr}$  and the result was independent of sweep rate at this rate.

### III. Results and Discussions

At least 3 PI-LB layers were needed to achieve good uniform LC alignment on PI-LB/ITO glass. The reason may be that 3 layers are enough to screen the interaction between the LC and ITO glass surfaces, and smoothly and fully cover the surface. All LC cells made with 3 to 15 PI-LB layers gave identical, good homogenous alignment. In all cases, the LC alignment direction matched with the dipping direction of LB deposition. To check the effectiveness of alignment of the last PI-LB layer, we deposited 6 layers on glass plates in such way that the dipping direction of the sixth layer was changed by  $45^{\circ}$  with respect to the dipping direction of the previous five layers. Polarized microscopy showed that the LC anchoring direction was solely determined by the dipping direction of the sixth PI layer, which means that the interaction responsible for LC alignment on PI-LB films is rather short ranged, i.e, less than the thickness of one PI layer ( $\sim 4.5\text{\AA}$  as measured by our group<sup>[15]</sup>). As is known, LC alignment on a PI-LB film is induced by the well ordered polymer, which aligns along the dipping direction<sup>[16]</sup>. The dipping speed is expected to be an important parameter for control-

ling the quality of the LB film. The influence of dipping speed and creep time on the order of PI-LB films was investigated by measuring the optical retardation. The result is shown in Fig.3. Because of the birefringence of the ITO glass itself, microscope slides were used. Assuming that the retardation  $R = 2\pi d_0(n_e - n_o)S/\lambda$ , where S is an appropriately defined orientational order parameter of the PI-LB film, and  $n_e$  and  $n_o$  are the extraordinary and ordinary refractive indices of the polymer respectively, it seems that in our range of dipping speed, the order of the LB film doesn't change. Furthermore, we did not see any difference in retardation of the PI-LB films for creep times of 1 hour and 2 hours.

The pretilt angles of LC cells with various numbers of LB layers were measured by the standard crystal rotation method<sup>[12]</sup>. The results are presented in Fig.4. All samples show very small or zero pretilt angle. When we applied an electric field across these cells, all of them showed reverse tilt domains under polarized microscopy, which confirmed the tilt angle measurement results. Introducing a finite pretilt angle will be very important for application of LB films as alignment layers.

Fig.5 shows two typical  $R/R_0$  versus  $1/CV$  curves of a liquid crystal cell with five PI-LB layers at two different temperatures. Fitting data of this type to Eqn.(1), the polar anchoring strength and extrapolation length at different temperature can be calculated and is shown in Fig.6. The polar anchoring strength of the PI-LB film is much stronger than that of  $SiO_x$ <sup>[13,14]</sup>, but somewhat weaker than that of a spin coated rubbed polymer film<sup>[3]</sup>.

As mentioned above, PI-LB films fail to align the liquid crystal with a pretilt angle, which is a prerequisite for LCDs to avoid reverse tilt domains. One technique to introduce a pretilt angle is to manipulate the side chain density of the polyimide precursor. The number of side chains can be controlled by varying the degree of imidization. It has been claimed that in this way that one can control the pretilt angle of the liquid crystal on such LB films<sup>[18]</sup>. However, so far, we don't understand how to force the side chains to flip down unidirectionally to give pretilt angles that are uniform over the cell. Furthermore, the thermal stability of the pretilt angle has not been investigated. An alternative method to get a finite pretilt angle is to rub the PI-LB film. Due to the fact that PI-LB films

are very thin (about  $22\text{\AA}$  for 5 layers), static charge accumulation should not be a problem. Fig.7 shows two crystal rotation curves of LC cells assembled with PI-LB films with and without rubbing. Clearly, the symmetry point shifted away from zero angle after rubbing. The pretilt angle obtained in this case was about  $2^\circ$ . For comparison, we also measured the polar anchoring strength of the rubbed PI-LB films, which is shown in Fig.8. It seems that rubbing doesn't enhance the polar anchoring strength of PI-LB films, which is not in agreement with the results of D.-S.Seo et al<sup>[8]</sup>. We also notice that the polar anchoring strength of our non-rubbed PI-LB film is almost the same as that of their rubbed one. Atomic force microscopy(AFM) images indicate that our PI-LB films are already highly ordered before rubbing<sup>[15]</sup>. Evidently rubbing does not further improve the order of the film. Based on these results, we suggest that the polymer backbones lay statistically flat on the glass surface along the dipping direction, and, therefore, they fail to align the LC with a pretilt angle. The stress generated by the rubbing process apparently gives these backbones a small but finite angle with the surface plane and a finite pretilt angle is obtained.

This result is consistent with the pretilt generation model proposed by J. M. Geary et. al<sup>[19]</sup>. However, it has to be mentioned here that it is hard to rub PI-LB films uniformly because of their ultrathin nature. Therefore, adjusting the rubbing strength is not a suitable way to control the pretilt angle for practical applications. We also find that hard rubbing can damage or even strip away PI-LB films. Controlling the pretilt angle of LC's aligned by PI-LB films is still a big challenge for the application of LB films in the LCD industry. Pretransitional birefringence has been used to probe the mechanism of LC alignment for some years<sup>[20-23]</sup>. We report here the first surface induced pretransitional birefringence measurements of a LC cell with PI-LB films as alignment layers. Three samples were measured and results are presented in Fig.9. The alignment layers for these samples are a PI-LB(5) film, a rubbed PI-LB(5) film and a conventional spin-coated rubbed PI film. All three samples show qualitatively similar pretransitional behavior. The solid lines in Fig.9 are fitting curves using Eqn.(3) derived from the Ginzburg-Landau theory of de Gennes.

Under the framework of de Gennes' theory<sup>[24]</sup>, the free energy den-

sity of a semi-infinite ( $z \geq 0$ ) sample near the LC isotropic to nematic phase transition is

$$F = F_0 + \frac{1}{2}a(T - T^*)Q^2(z) - \frac{1}{3}bQ^3(z) + \frac{1}{4}cQ^4(z) + \frac{1}{2}L|\nabla Q(z)|^2 \quad (2)$$

Minimizing the total free energy with respect to the functional form of  $Q(z)$  at fixed boundary condition  $Q(0)=Q_s$ , the analytic expression for total phase difference between ordinary and extraordinary rays of light passing normally through the sample is<sup>[22]</sup>

$$\begin{aligned} \Delta\delta &= \frac{2\pi}{\lambda}(n_e - n_o) \int_0^\infty Q(z)dz \\ &= \frac{2\pi}{\lambda}(n_e - n_o) \left(\frac{2L}{c}\right)^{1/2} \ln P(T, Q_s) \\ &= \delta_0 \ln P(T, Q_s) \end{aligned} \quad (3)$$

where

$$P(T, Q_s) = \frac{(F_1(Q_s)/Q_s^2)^{1/2} + (a(T - T^*)/2)^{1/2} + Q_s c^{1/2}/2}{(F_1(Q_s)/Q_s^2)^{1/2} + (a(T - T^*)/2)^{1/2} - Q_s c^{1/2}/2}$$

$$F_1(Q) = a(T - T^*)Q^2(z)/2 - bQ^3(z)/3 + cQ^4(z)/4$$

The parameters a,b and c for 5CB used for all fitting were  $0.13 \times 10^6 J m^{-3} K^{-1}$ ,  $1.6 \times 10^6 J m^{-3}$  and  $3.9 \times 10^6 J m^{-3}$  respectively and were obtained from the ref[23]. We selected  $Q_s$ ,  $T^*$ ,  $\delta_0$  and the background birefringence as adjustable fitting parameters to minimize the standard deviation.

Surprisingly, for all three cases, we found almost the same surface order parameters. The fitted values of  $Q_s$  were 0.38, 0.39 and 0.38 respectively for PI-LB(5), rubbed PI-LB(5) and spin coated rubbed PI films. The fitting results for the PI-LB film and rubbed spin coated PI film are fairly good. However, we notice that we can't fit both the high and low temperature regions for the rubbed PI-LB film. The magnitude  $\delta_o$  for the rubbed PI-LB film is 0.33, which is quite different from values 0.70 and 0.92 we got for PI-LB(5) and rubbed PI films respectively. They are supposed to be the same in the model because the same LC materials are used(see Eqn.(3)). Note that the data for this film fit de Gennes' model rather poorly by comparison with the other two films. This may indicate that the surface order parameter of the rubbed PI-LB(5) film changes with temperature instead of being fixed as we have assumed or that the uniformity of the rubbed PI-LB film is poor. More detailed work on this is under way.

The nucleation of the nematic phase of homogeneous LC cells with PI-LB(5) as alignment layers was observed under polarized light microscopy as the cell cooled slowly. As described in ref.[19], "sheet nu-

cleation" on the cell surface was observed as the nematic phase was entered. These uniformly aligned nematic layers grew in thickness until they met in the center of the cell, producing a flaw-free aligned texture. This behavior is strongly distinguished from the evolution of the alignment texture of a LC cell associated with obliquely evaporated  $SiO_x$  alignment layers. In the latter situation, the nematic phase nucleates at numerous isolated sites in the bulk and at the surface. These spots grow and join together with a high density of director defects. Uniform alignment of the whole cell is reached only after the defects anneal out. The PI-LB alignment behavior exactly belongs to the first category in table II of Ref.[19], i.e., sheet rather than point nucleation. On the other hand, obliquely deposited  $SiO_x$  alignment surfaces belong to the second category and are strong candidates for description by the Berreman model<sup>[25]</sup>. This is further substantiated by their failure to give pretransitional birefringence<sup>[21]</sup>.

Historically, the groove model was first proposed to elucidate the LC alignment mechanism for rubbed or polished solid surfaces<sup>[25]</sup>. Berreman, however, was well aware that surfaces having oriented

long organic molecules may align by a different mechanism. Much evidence shows that the groove structures induced by the rubbing process play a minor role in LC alignment on rubbed PI or many other polymer surfaces<sup>[19,27,28]</sup>. An epitaxial growth model was subsequently proposed in which the interaction between oriented polymers and LC molecules is responsible for LC alignment<sup>[19]</sup>. The mechanism of alignment is analogous to epitaxial formation of conventional solid crystals except it is orientational rather than translational in nature. However, to some extent there is still confusion about these two models. All of the above experimental results taken collectively provide evidence to settle this issue in favor of epitaxial alignment. This conclusion is independent of any specific macroscopic model such as that of de Gennes<sup>[24]</sup>.

We studied the case of LB films with a varying different number of layers deposited perpendicular to the direction of rubbing on a spin coated rubbed PI film. We also found that three PI-LB layers( $\sim 13\text{\AA}$ ) were required to switch the LC orientation from the rubbing direction to the PI-LB dipping direction<sup>[7]</sup>. Before deposition, well defined grooves were detected by AFM. The depth of

grooves was around 80Å. As we mentioned above, 3 PI-LB layers were also required to achieve good LC alignment on ITO coated glass. It seems that it also requires three PI-LB layers to screen the interaction between the LC and the polymer on spin-coated rubbed PI films. The groove stucture was still observed by AFM. After 3 layer PI-LB film deposition This result shows, not surprisingly, that while the groove structure may aid alignment, it is not an intrinsic reason for LC alignment on a rubbed polymer surface. Note also that groves on the rubbed polymer surface increases the interaction area between polymer and LC molecules in comparison with a PI-LB film with a flat surface. This could be the reason why the polar anchoring strength of a PI-LB film is lower than that of the rubbed PI film. Whatever the true effect of grooves may be, when the interaction between oriented polymer and LC molecules is screened by three or more perpendicularly oriented PI-LB layers, the influence of groove structure is dramatically weakened.

#### IV. Conclusions

Highly ordered multilayer PI-LB films were successfully fabricated by the LB technique and showed a strong tendency to align liquid crystals. In order to achieve good alignment on ITO coated glass, three or more PI-LB layers were necessary. It was also found that the anchoring direction of the LC was solely determined by the dipping direction of the uppermost PI-LB layer. This means that the alignment comes from short range molecular interactions. In our range of dipping speeds, the orientational order of our PI-LB films was almost constant. Unfortunately, they failed to align the LC with a pretilt angle. Rubbing was found to solve this problem, in principle, but it was very hard to rub the whole region uniformly. The measured polar anchoring strength between the PI-LB film and 5CB is very strong( $\sim 3 \times 10^{-4} J/m^2$  at  $30^{\circ}C$ ). The alignment texture and pretransitional birefringence behavior of a LC cell with PI-LB alignment films clearly showed that the aligning LC layer grows epitaxially from the surface. In the case of PI-LB films deposited perpendicular to the rubbing direction of spin coated rubbed PI films, results indicated that while the groove structure generated

by the rubbing process was not the intrinsic reason for LC alignment on rubbed polymer surface, it may enhance the anchoring strength.

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## Figure Captions

Fig.1 Flow chart of the multilayer PI-LB film preparation

Fig.2 Experimental set-up for the optical phase retardation and capacitance measurements. All elements are self explanatory except the one labeled AOM which stands for Acoustic Optic Modulator

Fig.3 Phase retardation of PI-LB(5) film versus the dipping speed.

Both sides of the microscope sheet glass have 5 layers of PI-LB films

Fig.4 Pretilt angle versus the number n of PI-LB(n) layers

Fig.5 Typical  $R/R_0$  versus  $1/CV$  curves at two different temperatures

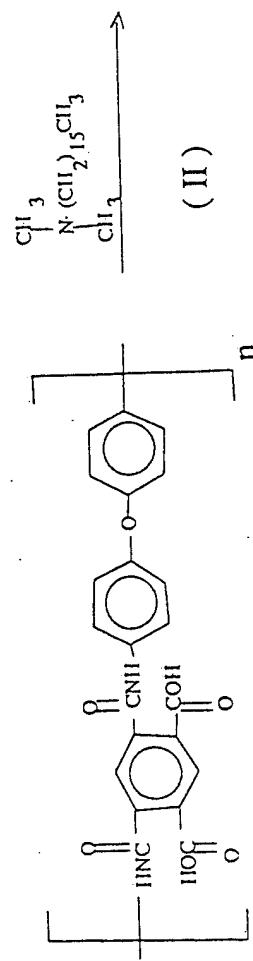
Fig.6 Polar anchoring energy and extrapolation length of PI-LB(5) versus temperature

Fig.7 Crystal rotation curves of LC cells having PI-LB(5) films with and without rubbing

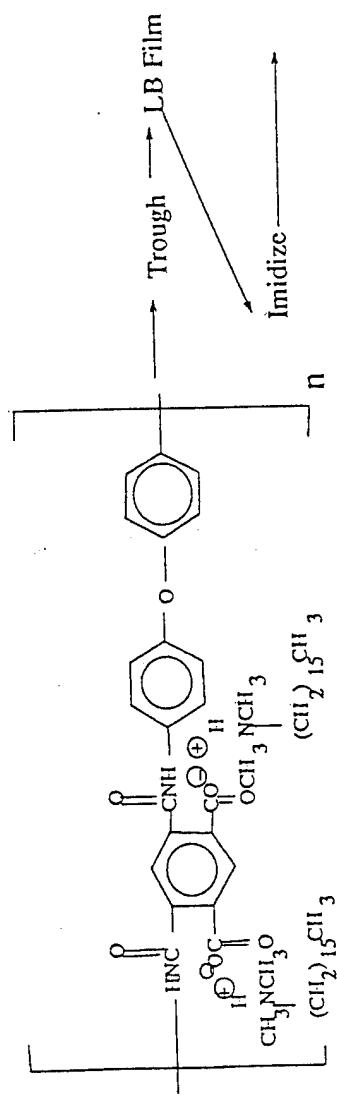
Fig.8 Polar anchoring strength and extrapolation length of rubbed PI-LB(5) versus temperature

Fig.9 Pretransitional birefringence behavior of LC cells assembled with PI-LB(5)(o), rubbed PI-LB(5)(▲) and spin coated rubbed PI(■) films. The solid curves are fitting curves using equation

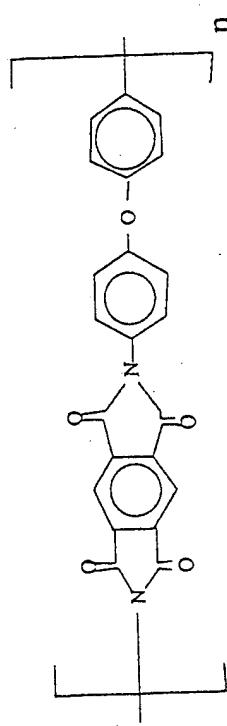
(3). Both experimental and theory values of the middle curve were shifted upward one degree on purpose in order to avoid overlapping the bottom curve



(1)

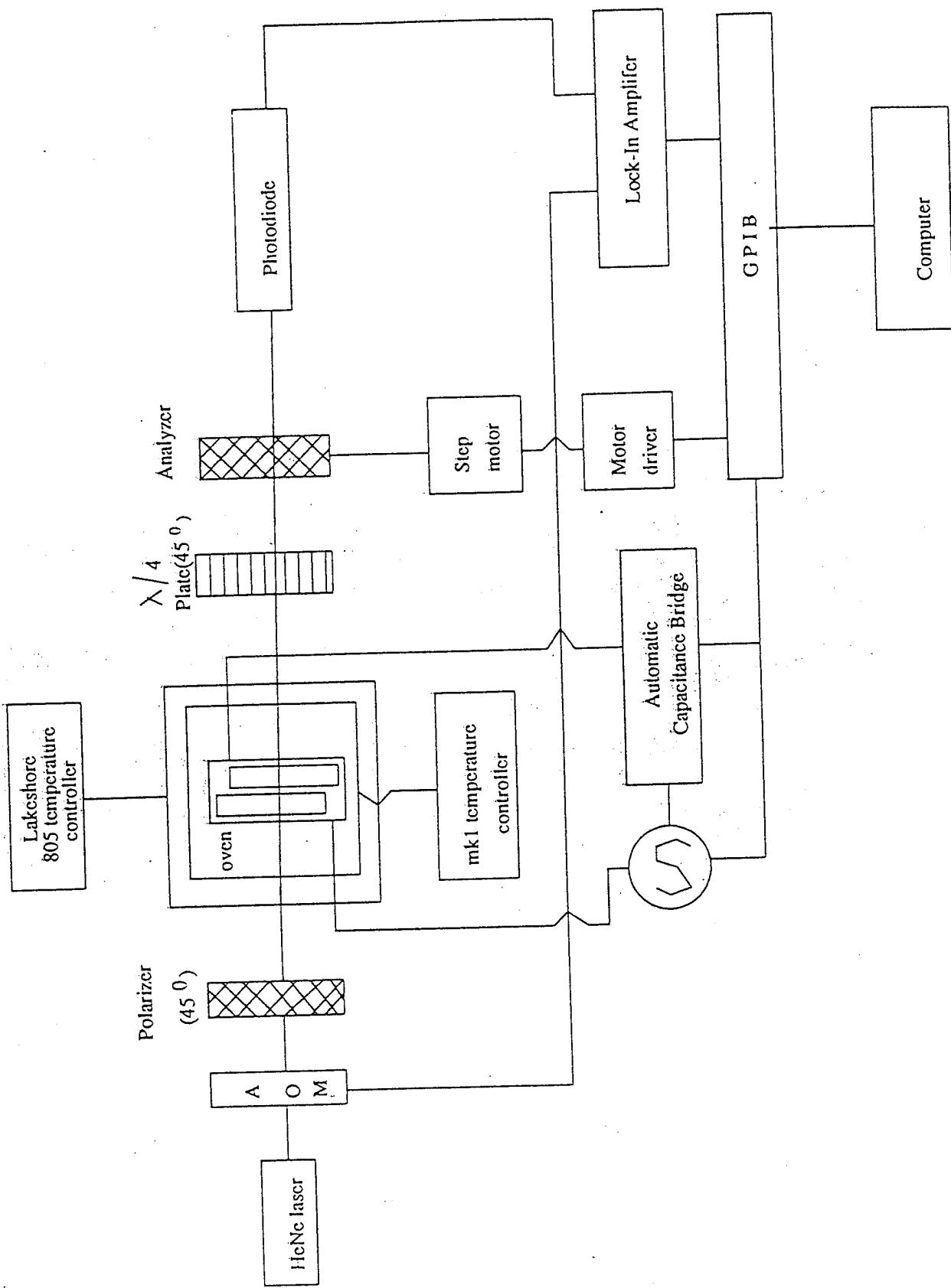


(III)



(IV)

151



T16.2

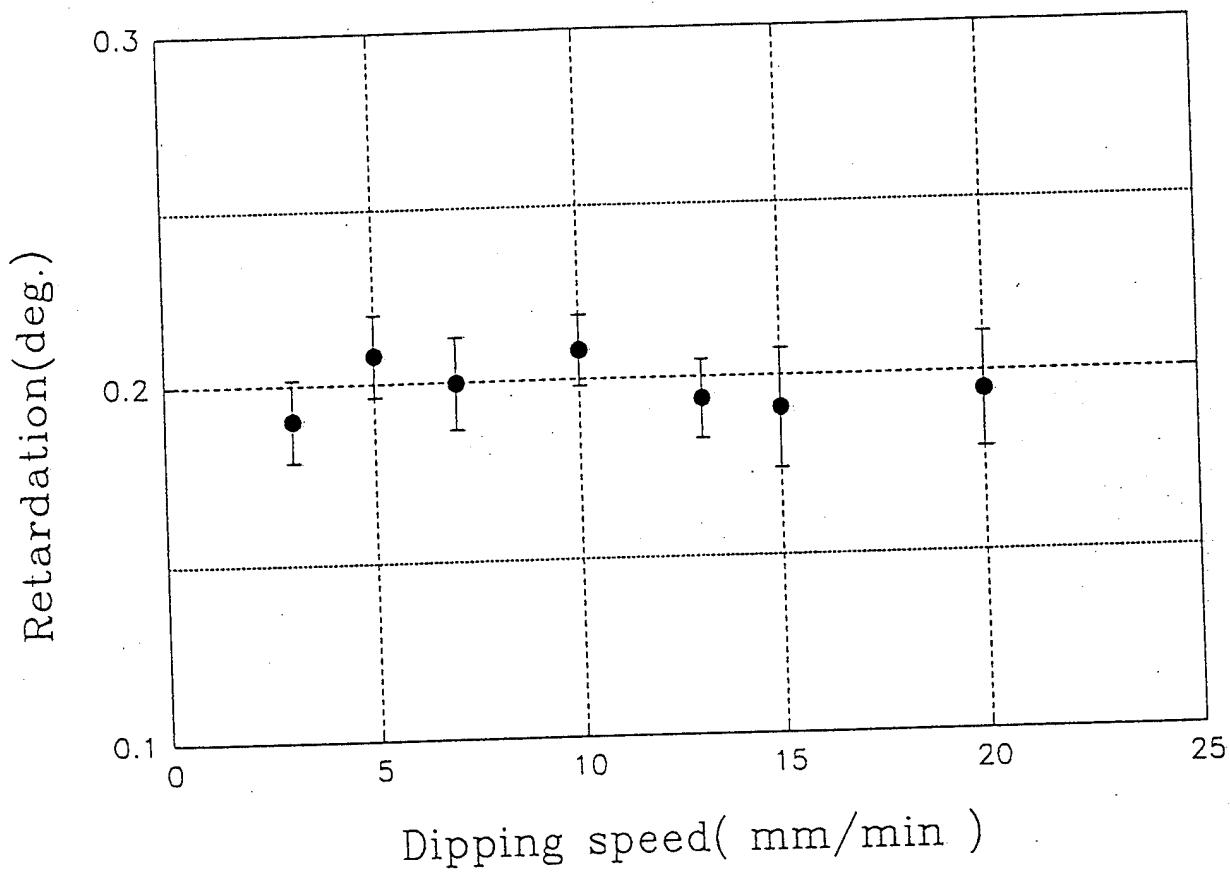


Fig.(3)

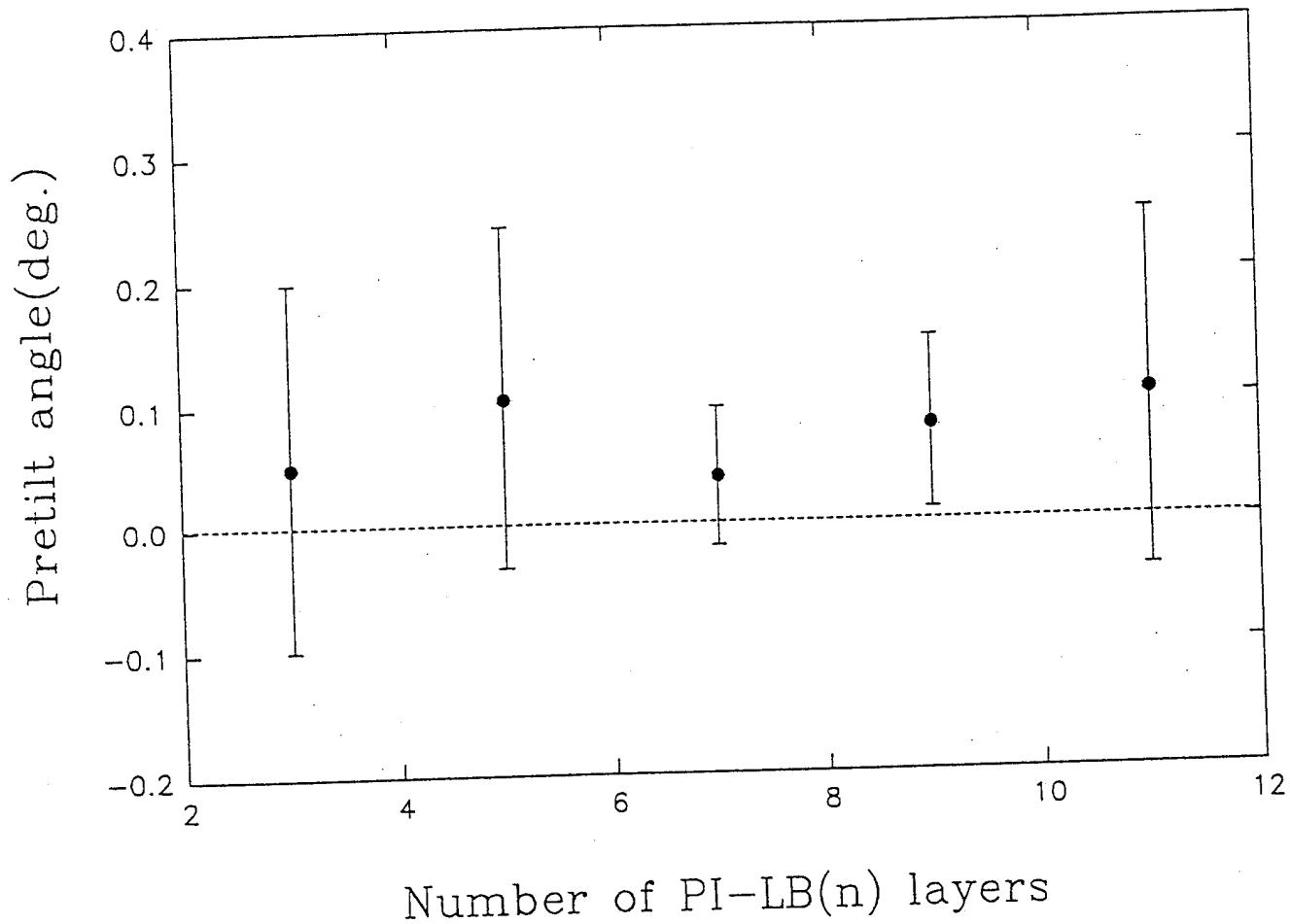


Fig.(4)

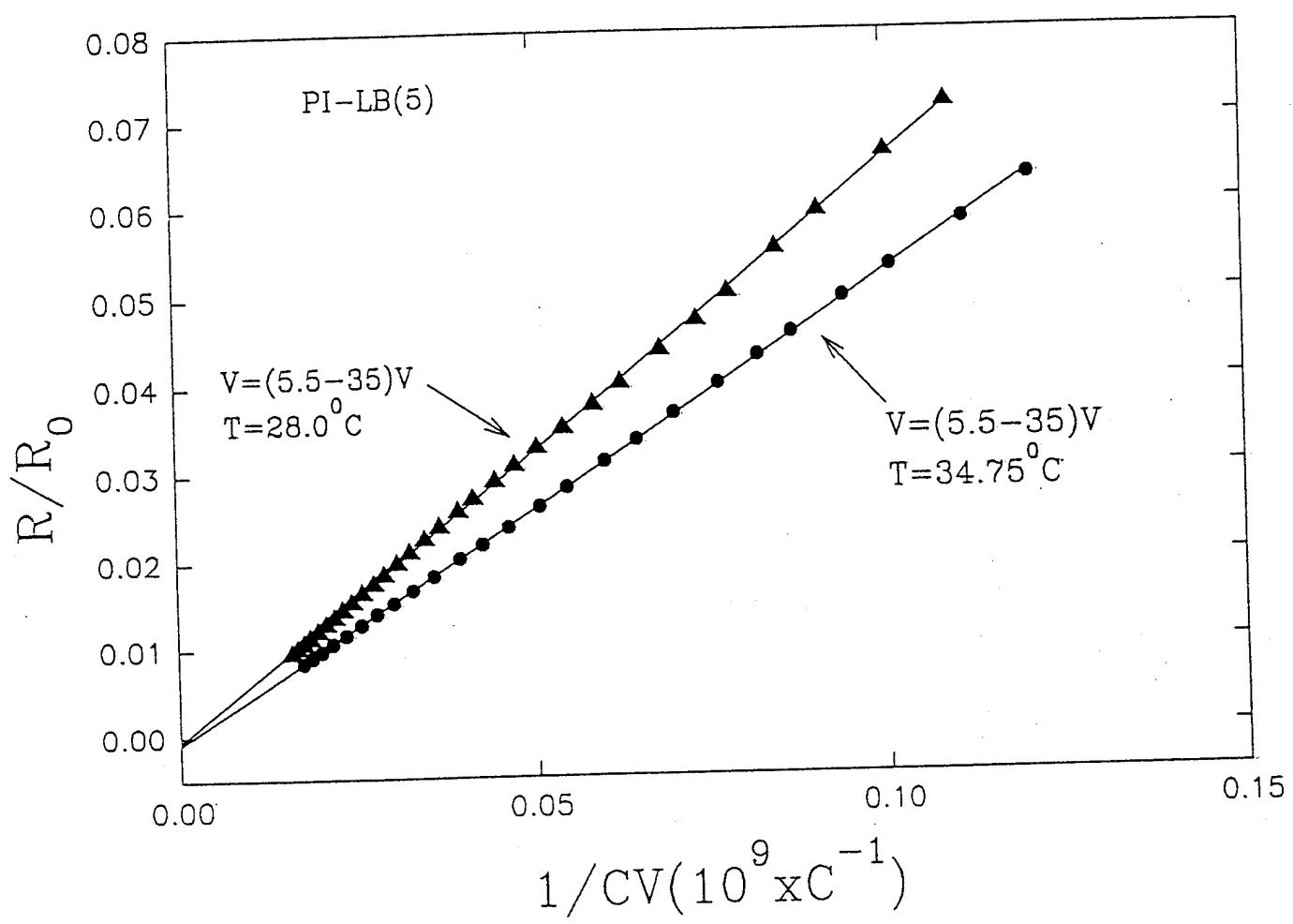


Fig.(5)

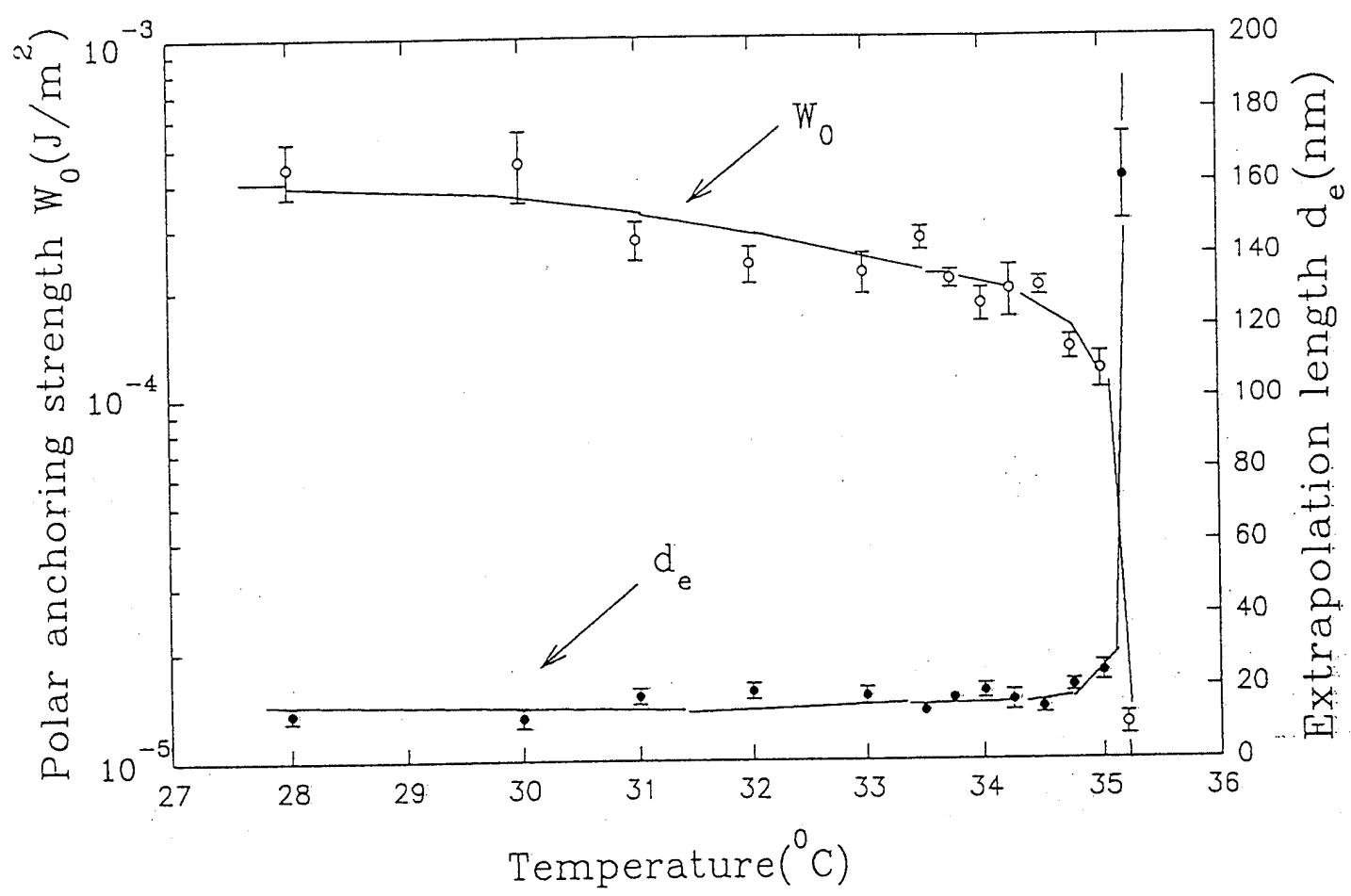


Fig.(6)

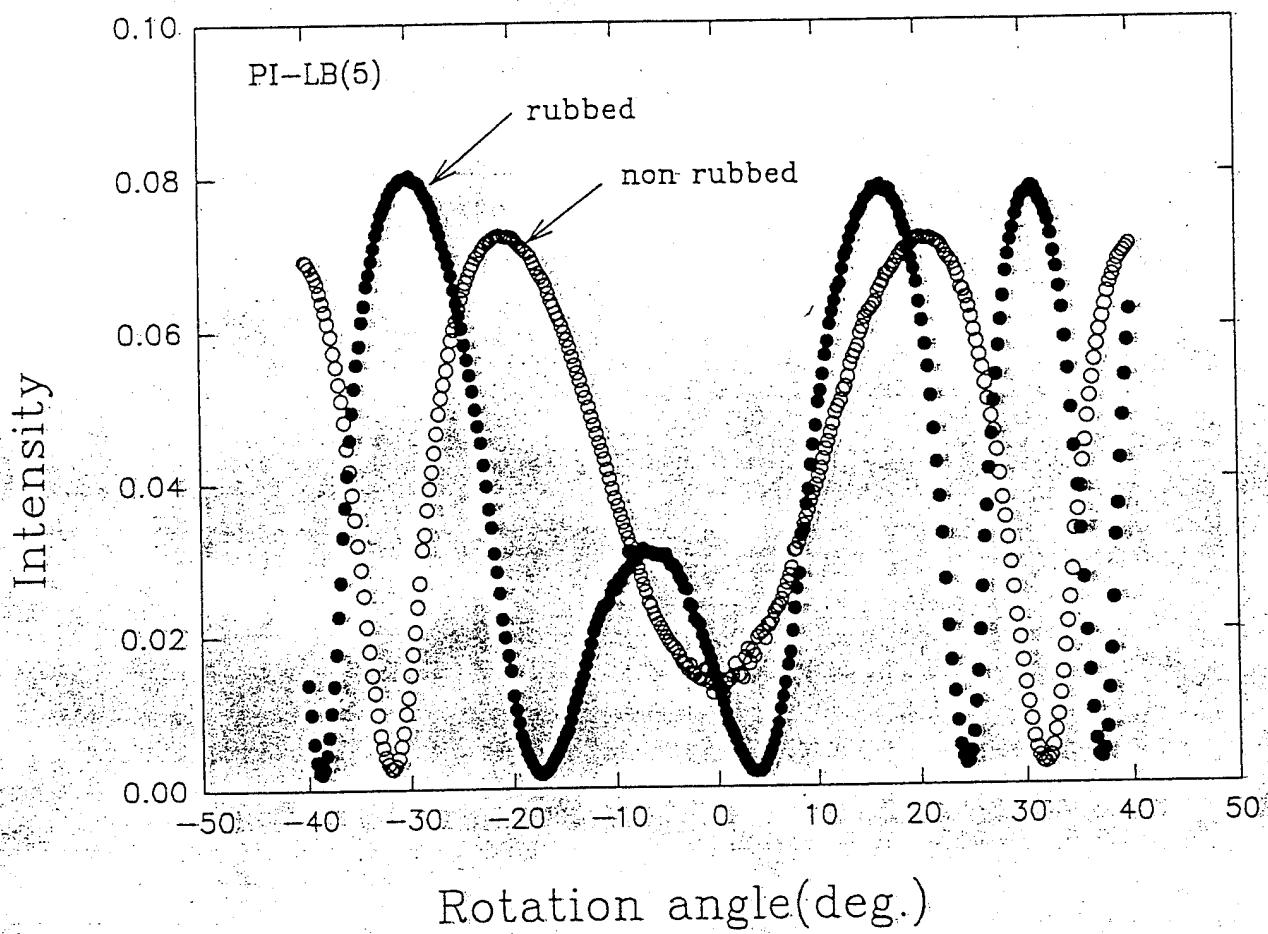


Fig.(7)

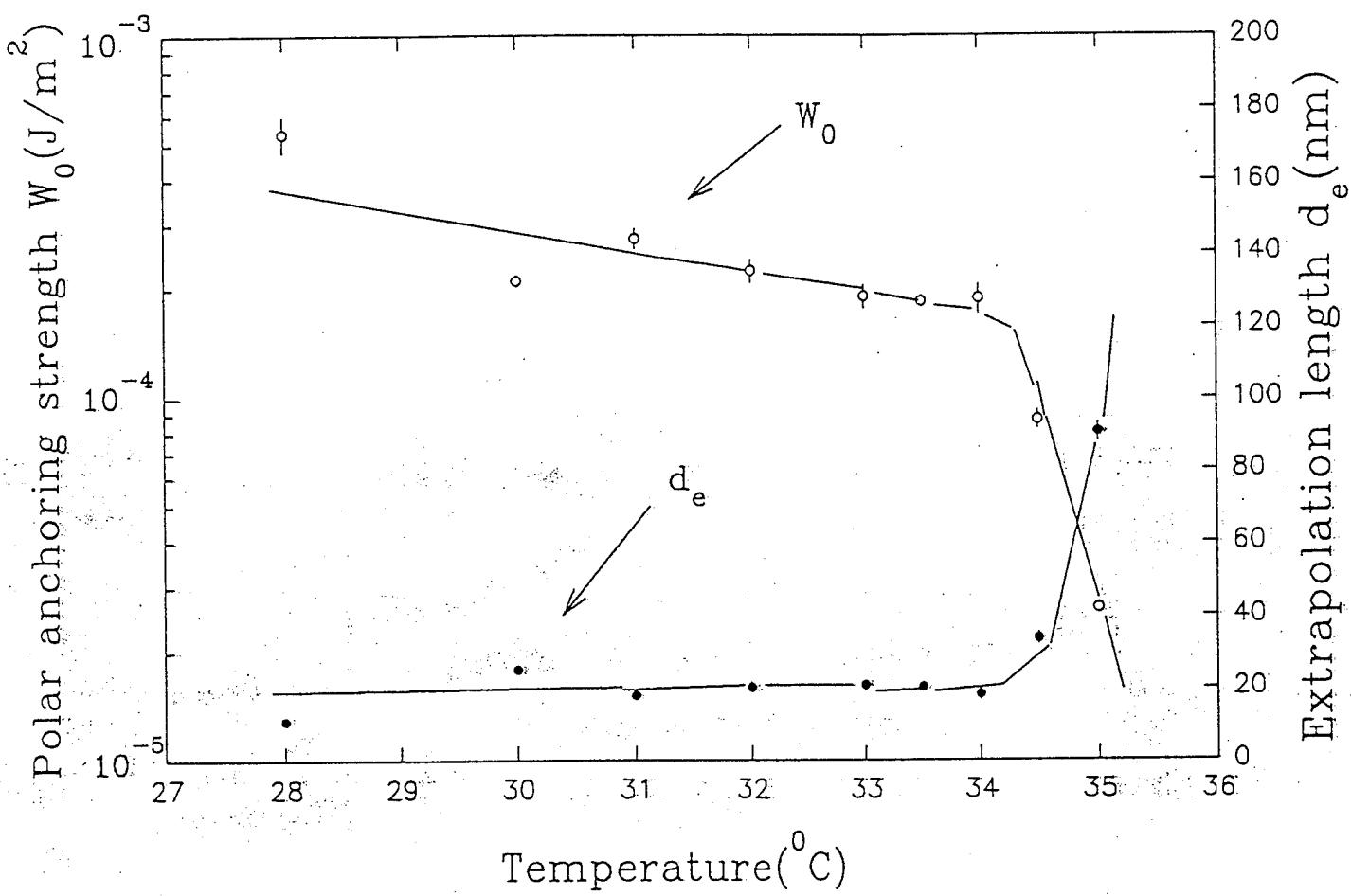


Fig.(8)

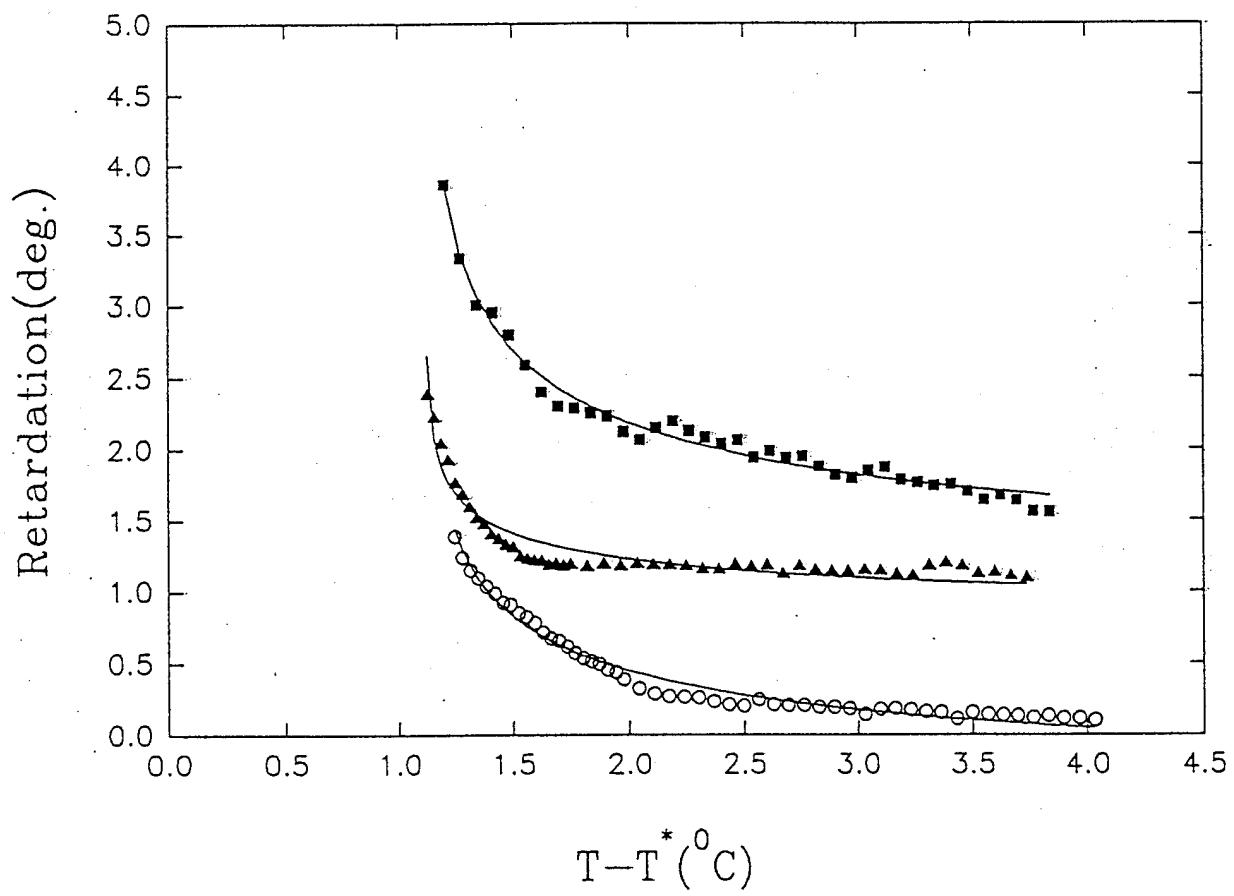


Fig.(9)